

INTRALAYER HYBRIDISATION TO COMBINE THE DUCTILITY OF SELF-REINFORCED POLYPROPYLENE WITH THE STIFFNESS OF CARBON FIBRE

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ABSTRACT

Combining a reasonable stiffness and a high ultimate failure strain in a single material is challenging task. Intralayer hybridisation of self-reinforced polypropylene (SRPP) with carbon fibre is proposed as a new strategy to increase stiffness while maintaining a high strain to failure. The bonding between SRPP and the carbon fibre prepreps was found to be a crucial parameter in these hybrids. It can be altered either directly by replacing the homopolymer polypropylene (PP) matrix in the prepreps by a maleic anhydride PP, or indirectly by changing the carbon fibre volume fraction. Weak bonding was key to preserving a high ultimate failure strain and impact resistance. Strong bonding reduced the ultimate failure strain and impact resistance, but improved flexural properties. These results reveal a delicate balance in optimising the bonding in hybrid composites to achieve optimum performance.

1 INTRODUCTION

Fibre-reinforced polymer composites face the stiffness-ductility dilemma: some composites are stiff but brittle, while others are ductile but compliant. Carbon fibre composites belong to the first category: they are stiff, but have a failure strain of 2% or less. Self-reinforced polypropylene (SRPP) belongs to the second category: it has a failure strain of 20%, but its stiffness is limited to 3-5 GPa for a woven composite. Examples of materials that are able to escape the stiffness-toughness dilemma exist in Nature. Silk fibre for example combines a stiffness of 5-60 GPa with a failure strain of 15-60% [1, 2]. These unique properties are attributed to its unique hierarchical microstructure. Unfortunately, silk fibres are very expensive, and its microstructure has not been successfully reproduced yet.

Fibre-hybridisation offers a way out of this dilemma. By combining two fibres in a single matrix, it is possible to combine the advantages of both fibres, while reducing some of their disadvantages. Three configurations exist for combining both fibre types: interlayer, intralayer and intrayarn. Interlayer hybrids consist of stacking layers of the two fibre types onto each other. In this configuration, each layer contains only one type of fibre, whereas intralayer hybrids have at least two fibre types within the same layer. The intrayarn configuration consists of two fibre types that are mixed on the fibre level, typically as co-mingled yarns. The interlayer configuration is the most common configuration. In literature on hybrid composites, it is generally accepted that well-dispersed fibres lead to better mechanical properties [3, 4]. The intralayer and intrayarn configuration should therefore lead to improved performance. Unfortunately, intrayarn hybrids are difficult to obtain commercially, as well-dispersed yarns are scarce [5]. The intralayer configuration therefore may

provide the optimal combination between availability and mechanical performance. It can be achieved using traditional weaving techniques with two dissimilar yarns. Several authors have already made direct comparisons between intralayer and interlayer hybrids, and confirmed the benefits of intralayer hybrids [6, 7].

The failure strain improvements in hybrid composites are limited by the failure strains of the hybridised fibres. The most common combination is carbon and glass fibre [3, 4]. Even though the failure strain of glass fibre is higher than that of carbon fibre, it is still limited to 3-5%. Hybridisation of a brittle and a ductile fibre is relatively new, and can lead to much larger improvements in the failure strain. Hybrids of SRPP, which consists of highly oriented PP tapes in a PP matrix, and carbon fibre can reach ultimate failure strains of 20% [8]. The challenge lies in developing suitable strategies for limiting the introduction of damage into the SRPP when the carbon fibres fail.

A vital parameter in hybrid composites is the bonding. Bunsell and Harris [9] for example showed the difference between a carbon/glass interlayer hybrids with good bonding and no bonding between the layers. In the unbonded case, the failure of carbon fibre was accompanied by a vertical load drop, whereas the bonded case led to a more gradual transfer of the load to the glass fibre layers. Similarly, Czél, Jalalvand and Wisnom [10, 11] have clearly demonstrated the importance of the mode II fracture toughness for controlling the damage mechanisms in hybrid composites.

Recently, Swolfs et al. [8] highlighted the importance of bonding in carbon fibre/SRPP hybrids. When a matrix film was added, the bonding improved, which prevented the damage from spreading over the sample and caused a drastic reduction in the ultimate failure strain. The addition of more carbon fibre prepregs may have a similar effect as adding matrix films. Most thermoplastic carbon fibre prepregs however contain excess of matrix, which locally increases the matrix fraction. This matrix fraction is known to be crucial in the bonding of SRPP [12, 13]. The goal of this study is to investigate two different prepreg types and analyse the link between their volume fraction, the bonding and the mechanical properties.

2 MATERIALS AND METHODS

2.1 Materials

Drawn polypropylene (PP) tapes were provided by Propex Fabrics GmbH (Germany). These homopolymer tapes have a draw ratio of 10-15, resulting in a stiffness of 10 GPa and strength of 500 MPa [14]. Their thickness and width are 50 μm and 2.4 mm respectively. A 20 μm isotropic film made from the same PP grade was provided as well.

CF prepregs were sourced from Jonam Composites (UK) and Mitsuya (Japan). The Jonam prepregs are 3 mm wide, 160 μm thick and contain T700S fibres in a homopolymer PP matrix. The Mitsuya prepregs are 5 mm wide, 60 μm thick and contain TR50S fibres in a maleic anhydride (MA) grafted PP matrix. The percentage of MA is unknown. These CF prepregs are referred to as CFPP and CFMAPP, respectively. The tensile strength of both carbon fibres is 4900 MPa, but the tensile modulus of the TR50S fibre (240 GPa) is slightly higher than that of the T700S fibre (230 GPa). The fibre volume fractions of the CFPP and CFMAPP prepregs are $32\% \pm 1\%$ and $46\% \pm 2\%$, respectively. Their microstructures are shown in Figure 1. Figure 1a reveals resin-rich outer regions for the CFPP prepregs, whereas they are absent for the CFMAPP prepregs in Figure 1b.

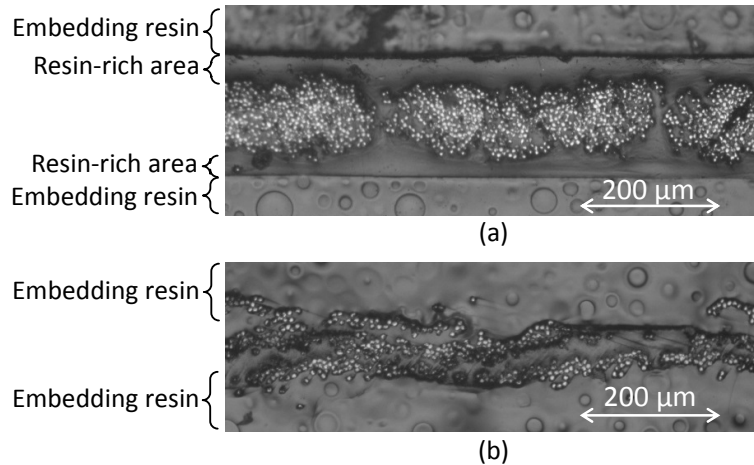


Figure 1: Microstructures of (a) CFPP prepregs, and (b) CFMAPP prepregs. The boundary between prepreg and embedding material is not always clearly visible for the CFMAPP prepregs.

The CF prepregs were co-woven with PP tapes by Propex Fabrics GmbH (Germany) in different ratios. All warp yarns were PP tapes, while the weft directions contained both PP tapes and CF prepregs. For the CFPP hybrids, 1 out of 13, 1 out of 7 and 1 out of 3 of the PP tapes were replaced by CFPP prepregs. These hybrids will be referred to as 3%, 7% and 11%, respectively, as this refers to their carbon fibre V_f (see Table 1). For the CFMAPP hybrids, 1/8 and 1/3 of the PP tapes were replaced by CFMAPP prepregs. These hybrids are referred to as 7%MA and 16%MA, respectively (see Table 1).

Type of hybrid	Label	Carbon fibre V_f	Sample thickness (mm)
Non-hybrid	0%	0%	1.23 ± 0.02
CFPP	3%	$3.4\% \pm 0.1\%$	1.34 ± 0.01
	7%	$6.9\% \pm 0.2\%$	1.47 ± 0.02
	11%	$11.0\% \pm 0.4\%$	1.67 ± 0.01
	7%MA	$7.0\% \pm 0.2\%$	1.40 ± 0.02
CFMAPP	16%MA	$15.7\% \pm 0.6\%$	1.96 ± 0.02

Table 1: Carbon fibre volume fractions and sample thicknesses for all hybrids.

The PP tapes were woven into a twill 2/2 pattern, but the CF prepregs were only interlaced with this pattern every four tapes. This places the CF prepregs more towards one side of the cloth, similar to a sateen weave. This allows the CF prepregs to be further away from the neutral line, which can be exploited for improved flexural properties. The fewer interlacing points reduces the crimp of the CF prepregs, which should lead to better surface quality and mechanical performance.

2.2 Hot compaction

The hybrid cloths were stacked in a (0/90/0/90)_s layup. The weft direction is labelled as the 0° direction as it contains the CF prepregs. The hybrid cloths were always oriented with the CF prepregs towards the outside. PP films were not added in between the layers.

The layup was placed in between two 1 mm thick aluminium plates. The press was preheated at 188°C for 10 min prior to inserting the layup to ensure a homogeneous temperature distribution over the press platens. After the layup was inserted, a pressure of 39 bar was applied for 5 min. Then, the layup was cooled down to 40°C in about 5 min, while maintaining the pressure.

2.3 Peel strength tests

T-peel strength tests according to ASTM D1876 were performed to measure the bonding in these intralayer hybrids. This test actually evaluates the interlayer bonding between two intralayer hybridised layers instead of the intralayer bonding. Intralayer bonding controls the debonding of the CF prepregs from the surrounding PP tapes. Since direct measurements of the intralayer bonding are highly challenging, a strong correlation between intralayer bonding and peel strength is hypothesised. We will consistently refer to intralayer instead of interlayer bonding, as it is the key parameter determining the mechanical properties of intralayer hybrids.

It is important to note that the top and bottom of the co-woven cloths are different. The weave architecture causes the CF prepregs to be preferentially on one side of the cloth. Four co-woven cloths were therefore stacked with the CF prepreg side towards the middle of the layup, resulting in a symmetric layup. A 12 μm polyimide peel ply between the second and third cloth was hence in direct contact with the CF prepregs. It should be noted that the addition of CF prepregs increases the stiffness of the peel strength samples. For a correct peel strength test, however, the sample legs should be compliant. Therefore, the layers in the peel samples were oriented to have the CF prepregs perpendicular to the peeling direction. In this case, the stiffness of the legs is nearly the same as for SRPP. The hot compaction parameters were exactly the same as for the other samples.

A sharp knife was used to cut the samples to a width of 20 mm and a length of 300 mm. The nominal sample thickness was 0.6 mm. The length of the unbonded length was 76 mm. The two unbonded ends were pulled apart at a rate of 254 mm/min. The samples were tested at room temperature on an Instron 5943 tensile machine with a 1 kN load cell. The peel strength was defined as the average peel load per mm width of the sample. The average was calculated over the first 127 mm displacement after the initial load peak, and 10 or more samples were tested for each configuration. The samples were tested in random order to minimise systematic errors.

2.4 Tensile tests

Tensile tests were performed according to ASTM D3039. Tests were performed on an Instron 4505 tensile machine equipped with a 100 kN load cell and hydraulic grips. Rectangular samples of 250 x 25 mm were tested at a gauge length of 150 mm. The sample thickness is summarised in Table 1. Sandpaper was used as end-tabs to avoid slippage in the clamps. The applied strain rate was 5%/min. Four or more specimens were tested for each configuration.

The speckle pattern on the sample surface was tracked by a camera. Digital image correlation was then performed to obtain the average surface strain in the longitudinal direction. After the CF prepreg failure, the damaged sample surface prevented measurement of the strain. The approach described in Swolfs et al. [8, 15] was used to resolve this issue. The crosshead displacement of the tensile machine was used to calculate the strain after the carbon fibre failure. This strain was shifted by a constant factor to ensure strain continuity when the CF prepregs fail. The tensile modulus was calculated as the slope between strains of 0.1% and 0.3%.

2.5 Flexural tests

Three point flexural tests were performed according to the ASTM D790 standard. The span length was 60 mm for all samples, corresponding to an average span-to-thickness ratio of 40 and a minimal ratio of 30. The displacement rate was set to 4 mm/min, corresponding to a strain rate of 1%/min on average. The nominal sample length was 90 mm, while the nominal sample width was 20 mm instead of the recommended 10 mm. This reduced the scatter in the data due to the large unit cell of the hybrid cloths. A 10 mm width would cause significant variations in the number of carbon fibre yarns in each sample. At least five samples were tested, all of which had the outer layers in the 0° direction. The flexural modulus was calculated between 0.1 and 0.3% flexural strain.

2.6 Penetration impact tests

Falling weight impact tests were performed on a CEAST Fractovis 6789 machine according to ISO 6603-2. A hemispherical tup with a diameter of 20 mm was used. All samples were clamped at the maximum pressure of 9 bar, corresponding to a force of 5600 N. Sample sizes were 100x100 mm and at least six samples were tested for each hybrid. The load was registered by a 20 kN load cell in the tup, while a laser system measured the displacement.

The striker was set to a height of 1 m. The inner and outer diameter of the clamp were 40 and 60 mm, respectively. The mass of the striker was 26.17 kg, corresponding to a total energy of 257 J. This was sufficient to cause penetration in all samples. The energy absorption was calculated as the area underneath the load-displacement diagram.

3 RESULTS

3.1 Peel strength

The peel strength of non-hybrid SRPP increases by interleaving woven PP tape layers with PP films [13, 16]. Adding CF prepregs could also cause the intralayer bonding to increase, as the CF prepregs introduce additional matrix material to the hybrid.

The peel strength was proportional to the carbon fibre V_f for the CFPP and CFMAPP hybrids (see Figure 1). Please note that the observed increase cannot be attributed to the increased stiffness of the layers by having the prepregs perpendicular to the peeling direction. The maleic anhydride in the CFMAPP hybrids increased the peel strength by improving the adhesion of MAPP to the carbon fibres and the PP tapes. The peel strength increases notably faster with carbon fibre V_f for the CFMAPP hybrids than for the CFPP hybrids. The next step is to assess the influence of these differences in peel strength or intralayer bonding on the mechanical performance.

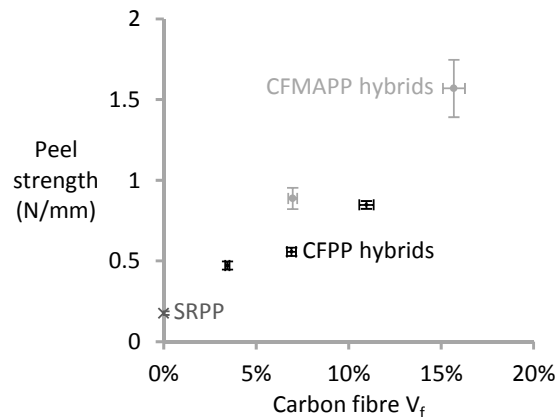


Figure 2: Peel strength of hybrid composites with CFPP and CFMAPP prepregs as a function of the carbon fibre V_f . The peel strength values for SRPP with and without PP films were added to facilitate comparison. These films were not used in the hybrids.

3.2 Tensile properties

In previous studies on carbon fibre/self-reinforced hybrid composites, the presence of matrix films reduced the ultimate failure strain in tension [8]. This was due to the increased intralayer bonding caused by these films. An increased carbon fibre V_f may therefore have a similar effect here. The tensile properties of the hybrid composites are shown in Figure 3. For the CFPP hybrids, the 20% ultimate failure strain of the non-hybrid SRPP was maintained up to a carbon fibre V_f of 7%. For the 11% hybrid however, the ultimate failure strain was reduced to around 6%. This reduction occurs faster for the CFMAPP hybrids, as they have a stronger bonding.

The strong intralayer bonding in the 7%MA hybrids decreased the ultimate failure strain, and this decrease is even stronger in the 16%MA hybrids. This is due to the higher peel strength with increased carbon fibre volume fraction (see Figure 2). High peel strengths indicate the CF prepregs are well bonded, which prevents the debonding along the CF prepregs from growing. This debonding is needed to prevent localisation of the strains in the debonded region, and hindering this debonding hence causes premature failure. Visual inspection revealed that regions in between the longitudinal carbon fibre prepregs are still bonded together after the tensile test. This confirms that the tensile behaviour is not controlled by delamination, but by debonding of the carbon fibre prepregs. The ultimate failure strain of these hybrids is therefore controlled by the intralayer instead of interlayer bonding. It should however be emphasised that both parameters are strongly correlated.

A strong intralayer bonding localises the strain in a certain region of the specimen, as it prevents further debonding. This can be observed on the specimens after the tensile test (see Figure 4). The 7% hybrid is completely debonded within the gauge length, whereas the 11% hybrid debonded only partially.

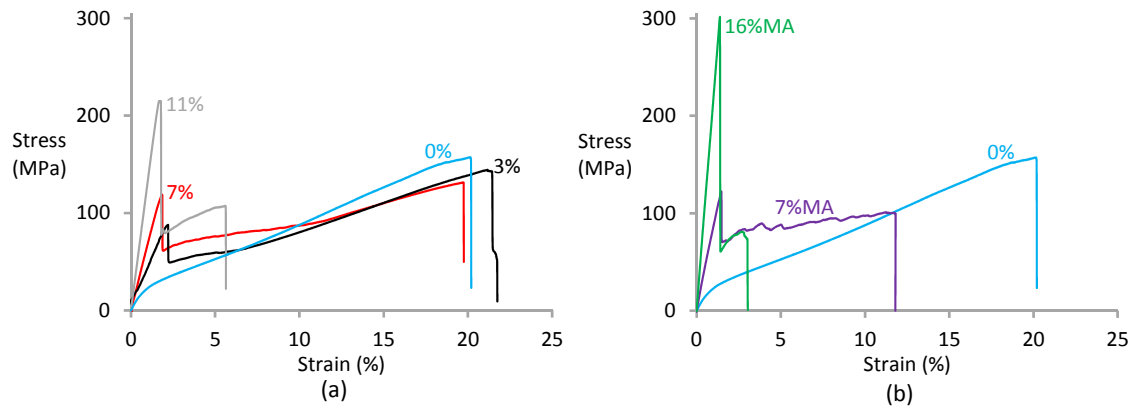


Figure 3: Representative tensile diagrams of the hybrid composites for different carbon fibre V_f : (a) for the CFPP hybrids, (b) for the CFMAPP hybrids.

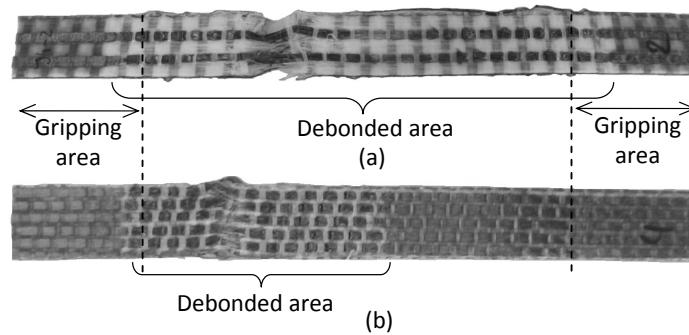


Figure 4: CFPP hybrid samples after a tensile test: (a) 7% hybrid, and (b) 11% hybrid. The debonded area extends slightly into the gripping area.

The tensile modulus of the CFPP hybrids is higher than that of the SRPP reference (see Figure 5). Nevertheless, the increase is not as pronounced as expected due to the presence of out-of-plane undulations in the carbon fibre prepregs. The increase is more pronounced for the CFMAPP hybrids, where these undulations were absent.

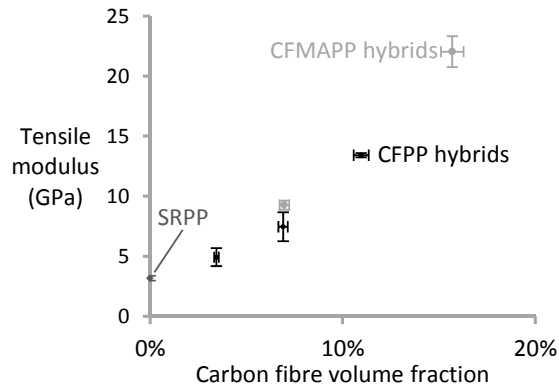


Figure 5: The tensile modulus of the CFPP hybrids increases slower with increased carbon fibre V_f than for the CFMAPP hybrids.

3.3 Flexural properties

The matrix type in the CF prepreps has a pronounced influence on the flexural behaviour of the hybrid composites (see Figure 6). Adding up to 7% of carbon fibre has only a small effect on the flexural behaviour. The 7%MA layup on the other hand shows a much stronger increase in stiffness and strength, despite having the same carbon fibre V_f . Large changes in the flexural behaviour of the CFPP hybrids only occur when the carbon fibre V_f is increased to 11%. Compared to the low flexural modulus of the 3% and 7%, the flexural modulus of the 11% layup is three times higher. This 11% layup has a high peel strength, similar to that of the 7%MA hybrids (see Figure 2).

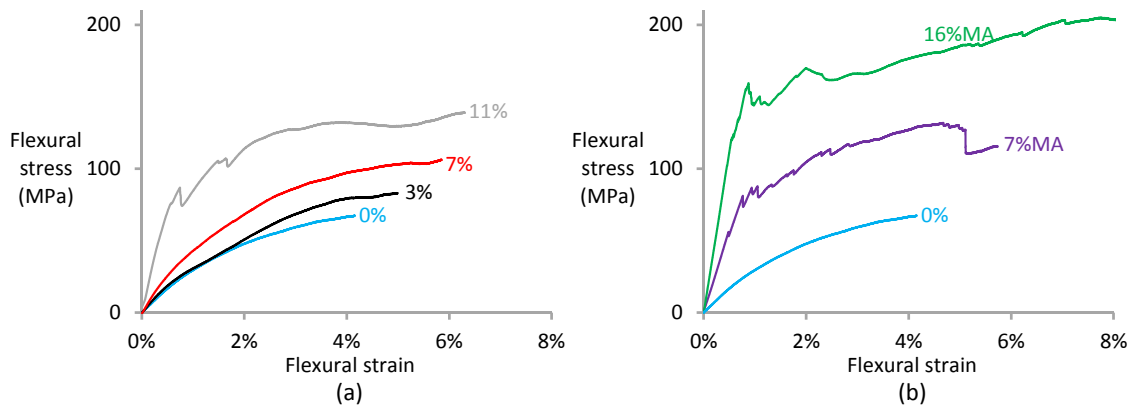


Figure 6: Flexural diagrams for the (a) CFPP hybrids, and (b) CFMAPP hybrids.

The flexural modulus of the 3% and 7% CFPP hybrids reveals only a modest modulus increase compared to SRPP (see Figure 7). The modulus increase is more pronounced for the CFMAPP hybrids. The slow increase for the CFPP hybrids is attributed to the presence of undulations. This essentially creates regions with pre-buckled fibres. Buckling in compression was experimentally observed within the strain interval for the flexural modulus calculations. This is attributed to the low adhesion of carbon fibre to PP. The presence of undulations in the CFPP hybrids make them use carbon fibre in a less efficient way than CFMAPP hybrids.

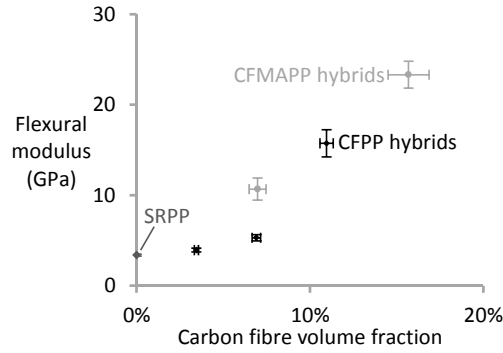


Figure 7: The flexural modulus of the CFPP hybrids increases slowly with increased carbon fibre V_f . The stronger bonding in the CFMAPP hybrids leads to a faster increase.

3.4 Impact resistance

Impact resistance is the major advantage of SRPP [17]. The addition of brittle carbon fibres is expected to reduce the impact resistance. Nevertheless, the aim is to minimise this reduction by an intelligent design of the hybrid composite.

For facilitating the comparison, the penetration impact resistance will be compared against a linear rule-of-mixtures. For the SRPP reference composite, a penetration impact resistance of 32 ± 3 J/mm was measured. For the all-carbon fibre reference composite, a reasonable estimate of 10 J/mm was chosen based on literature [18]. This value was assumed for CFPP and CFMAPP, even though the matrix and CF volume fraction was different.

The penetration impact resistance of the CFPP hybrids reduces by adding more carbon fibre, but this reduction is limited (see Figure 8a). The linear rule-of-mixtures yields reasonable predictions in this case. The penetration impact resistance of the CFMAPP hybrids, however, is much worse than for the CFPP hybrids (see Figure 8b). The linear rule-of-mixtures strongly overestimates the measured values for the CFMAPP hybrids. The strong bonding of the MAPP caused the composite fail in a brittle manner, by limiting the debonding, delamination and PP tape fibrillation. This creates local fracture, as illustrated in Figure 9b. The fracture is localised along the lines of a '+'-shape, indicating a rather brittle fracture. The damage in the four lips of the '+'-shaped fracture still have a stiff feeling, indicating that they were not debonded or delaminated. In CFPP hybrids however, the weak bonding of the PP facilitated debonding and delaminations. This created a fibrillated appearance (see Figure 9a) and a large portion of the specimen to absorb energy. The protruding parts of the penetrated specimens became compliant, indicating that they were delaminated and debonded. A large volume of material hence contributed to energy absorption during impact.

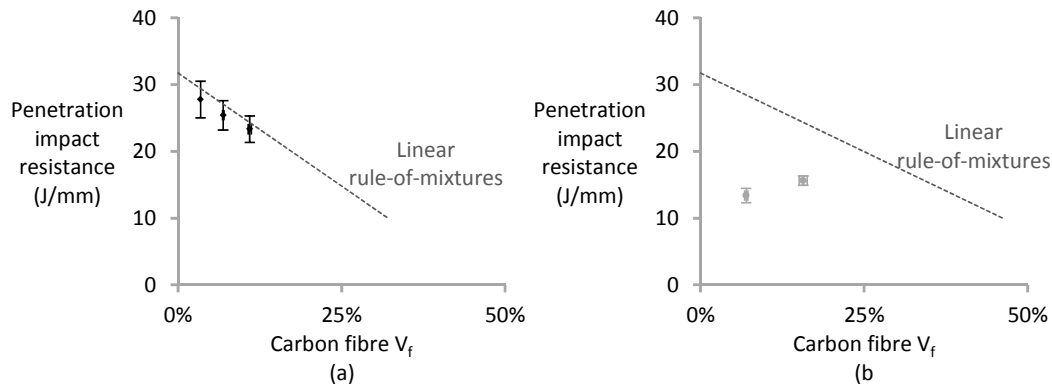


Figure 8: Penetration impact resistance for the hybrid composites, compared to the linear rule-of-mixtures.

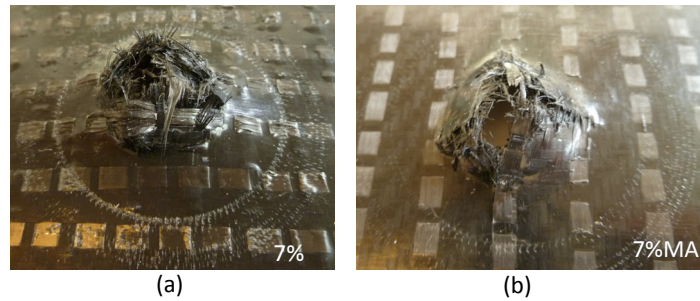


Figure 9: Sample appearance after penetration impact, showing extensive damage in (a) the 7% hybrid, but more limited damage in (b) the 7%MA hybrid.

4 CONCLUSIONS

Hybrid composites have the unique potential combine a reasonable stiffness with a high ultimate failure strain and impact resistance. This requires careful optimisation of the bonding. A weak bonding was required to maintain a high ultimate failure strain in tension. Peel strengths above 0.8 N/mm led to strong reductions in the ultimate failure strain. For penetration impact resistance, the peel strength was insufficient to explain the observed difference, as the matrix inside the preregs was found to be crucial. For MAPP preregs, the penetration impact resistance was strongly reduced, as energy absorbing mechanisms such as debonding and delamination was hindered too much. The CFPP hybrids however were able to maintain a high impact resistance.

A weak bonding may seem optimal for tension and impact resistance, but it also decreases the flexural properties. A good bonding is required to minimise the presence of out-of-plane undulations in the preregs. These undulations strongly reduce the flexural modulus and strength. This highlights the delicate balance of finding the right bonding level, which will depend on the application requirements.

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